STUDIES ON THE PRODUCTION OF CARBON BLACK FROM COAL

M.S. Iyengar, R. Haque, R. K. Chakrabarti and M. L. Dutta

Regional Research Laboratory, Jorhat, (Assam), India

ABSTRACT

Earlier work revealed that high volatile, vitrain rich and low ash coals could be converted to carbon black equivalent to thermal grade carbon black of commerce. In the present paper which is an extension of the earlier work some other coals have been used. It has been shown that by changing the reactor design and reaction variables the carbon black obtained is found to have properties of rubber and tyre grade products. Properties and compounding tests of the carbon black in rubber mix have been carried out and compared with those of standard products. A cost economics of the carbon black obtained from coal has also been discussed.

Coal has traditionally been used as fuels and there are very few studies describing the nonfuel use of coals. Johnson and co-workers (1,2) were the first to report on the conversion of high volatile coals into thermal black. They obtained the black as a byproduct in the production of hydrocyanic acid from coal. In an earlier paper (3) the present authors have described the production of thermal black from Assam coal. They adopted a transport reactor very much different to that used by the earlier workers and their main product was carbon black.

But thermal blacks find very limited use. The major demand is for rubber grade black for use in the tyre industry. The present paper describes studies on obtaining improved grades of carbon black from three bituminous coals of India. The paper also describes the techno-economic aspects of producing carbon black from coals.

Equipment and the process:

While the apparatus in the present studies are similar to that described earlier (3), the reactor, however, was designed to handle 70 lb./hour of coal. The inner wall of the reactor was made of 9 in. thick high alumina bricks followed by a 6 in. layer of insulation brick followed by a M.S. sheet. The reactor was 12 ft. high and 1 ft. in diameter.

A typical run was carried out as follows:-

Crushed coal (40 per cent through 200 BS) fed to the reactor through a rotary feeder is conveyed through the transport reactor by the carrier gas air. The reactor is initially heated to 500 - 600°C by burning low ash firewood. When the temperature becomes steady at the above temperature, the charging of coal is started. Partial combustion of the coal raises the temperature to 1300°C maximum. The air to coal ratio of the charges is kept constant at 65 cu.ft./lb. and the time of residence of the coal particles in the reactor is about 1.3 seconds. The carbon black is collected in the temperature range of 1200 - 1300°C. The char is separated from carbon black in a stainless steel cyclone separator. The carbon is recovered by first scrubbing it with water in the scrubber and then by electrostatic precipitation.

The product is then pelletised and dried at 400 - 450°C. Strict control of air/coal ratio and feed rate are important as changes may lead to existence of uncracked tar in the carbon black and deteriorate its property or lead to excessive combustion and give lower yield of the product. Figure 1 shows the unit for producing carbon black from coal.

Experimental results and discussion -

The work was carried out with three different coals of India formed under widely varying geological conditions. Table I shows the analysis of the coals used in the study.

Though they are all high volatile low rank coals, they differ in their caking characteristics and ash content. Sulphur content of the third coal is much lower than the two other coals. Reaction conditions were maintained identical and average test result of two or three runs has been shown. Table II shows the analysis of carbon blacks.

Figure 2 shows that the yield of carbon black bears a direct relationship with the hydrogen content of the coal. The Russians also obtained such relations (4), but details of their process are not available. The formation of carbon black from coal initially involves decomposition of coal to tar and gaseous hydrocarbons which ultimately undergoes dehydrogenation and aggregation to form carbon black. Theories on the carbon black formation differ mainly in the route and order in which these two essential changes occur (5).

Coals containing high mineral matter give a higher ash in the product. Consequently compounding tests have been carried out with only low ash carbon black produced from Baragolai colliery coal, Upper Assam. Figure 3 shows the electron micrographs of carbon black produced from Baragolai colliery coal (a) and HAF black(b). The study was carried out using a magnification of 52700 to determine the average particle size and the structure of the black. It is observed that carbon black obtained from coal is of high structure and is different from thermal black, which is also supported by its compounding test. The size of these particles varies from 200 - 800° A comparable with HAF blacks having particle size varying between 200 - 800°A. However, the frequency of distribution of bigger particles in the HAF black is much less than those in carbon black prepared from coal.

Figure 4 shows the x-ray pattern of carbon blacks obtained from (1) Baragolai colliery coal, (2) Pench Valley coal, and (3) Standard Sample HAF. It may be observed that the carbon blacks obtained from coals show bands of mineral matters, especially quartz, which is more prominent in the case of sample 2 obtained from Pench Valley coal containing higher percentage of mineral matter. The carbon band of samples 1 and 2 differ in intensity from that of number 3 as the formation of the latter takes place at temperature higher than those of 1 and 2. This may be attributed to the randomness in the degree of orientation of carbon particles in samples 1 and 2 whose formation takes place at lower temperature than that of sample 3.

Properties of the carbon black obtained from Baragolai colliery coal, Assam have been compared with two commercial carbon blacks in Table III.

Acetone extract which indicates the presence of uncracked tar in the product is slightly higher than those of the standard blacks. Iodine absorption values are greater than even HAF blacks. Surface area has been calculated from iodine absorption values (6), using the relationship SDd = 60,000, where S = surface area, D = density, d = particle size. D.B.P. absorption is very nearly equal to the HAF blacks but greater than the SRF blacks. pH of the product is in the mild acidic range which may be attributed to the nature of starting material.

Table IV shows the physical properties of rubber compounded with carbon black obtained from Baragolai colliery coal and compared with those compounded with a standard SRF black. Mooney viscosity which is an important property of the polymer mix is dependant mainly on the carbon black structure and loading in most polymers. In a 50 phr sample Mooney viscosity @ 100°C is found to be higher than the SRF blacks and Mooney scorch time about 0.64 times that of SRF blacks.

Rate of cure of the compound loaded with carbon black derived from coal is faster than compounds loaded with SRF blacks.

Physical properties like tensile strength, elongation at break and 300% modulus of the compound loaded with carbon black from coal are in line with SRF blacks, which shows it can substitute SRF blacks.

Cost economics:

A cost study of carbon black produced from coal revealed that the cost of production is \$ 70 per ton on the basis of 20 per cent conversion of coal to carbon black and price of coal being taken \$ 7.5 per ton. The price of char produced in the process (about 30 per cent) is taken as \$ 10.00 per ton. This is quite promising as the market price of carbon black in India varies from \$ 225 - 325 per ton.

Conclusion -

By changing the design and material of construction and altering the reaction variables the quality of the carbon black obtained from coal can be considerably improved to approach those of the rubber grade product. Some of the properties are similar to those of HAF. Though the physical properties like tensile strength, elongation at break and 300 per cent modulus of the compound loaded with carbon black from coal are lower than the compounds loaded with HAF blacks, they are in line with the SRF blacks. Experiments are still underway to produce HAF black.

Acknowledgement:

Grateful acknowledgement is made to M/s Synthetics and Chemicals Limited, Bombay and National Physical Laboratory, New Delhi for assistance and co-operation.

Literature Cited:

1. Johnson, G.E., Decker, W.A., Forney, A.J., Field, J.H. 158th National Meeting of American Chemical Soc. Division of Fuel Chemistry, New York, Sept. 7 - 12, 1969, Vol. 13, No. 3, pp.207-222.

2. Johnson, G.E., Deckar, W.A., Forney, A.J., Field W.H. Rubber World, 156(3), 63-68, 1967.

3. Haque, R., Chakrabarti, R.K., Dutta, M.L., Iyengar, M.S. Sent for publication in J. Ind. Eng. Chem. R & D Section (under review).

4. Mischenko, M.L., Bagdanov, I.F., Farberov, I.L., Urkovski, I.M., Gilyazetdinov, L.P., Gugaev, B.I. Khim, Tverd. Topl., (2), 139-141 (Mar-Apr., 1969).

5. Gaydon, A.G., "The Spectroscopy of flames". Chapman & Hall Ltd., London, 1957, p. 189.

6. Smith, W.R., Thornhill, F.S., Bray, R.I., Ind. Eng. Chem. Indust., 33, 1303 (1941).

Table I - Analysis of the coals used for production of carbon black.

Details of the coals	Ash %	Moisture %	V.M. % DAF	Fixed Carbon %	C % DAF	H % DAF	S % DAF	N+O by diff. % DAF	Caking properties
1. Baragolai colliery, Upper Assam	2.7	1.8	42.3	57.7	81.4	5.6	2.5	10.5	Highly caking.
2. Garo Hill, Lower Assam	6.5	1.4	49.5	50.5	80.3	5.8	2.5	11.4	Weakly caking.
3. Pench valley, M.P.	23.4	4.5	35.0	65.0	82.5	5.2	0.7	11.6	Non-caking.

Table II - Analysis of carbon blacks produced from different coals under reaction conditions.

Coal feed rate - 70 lb,/hr.

Air/coal ratio - 65 cu. ft./lb.

Temperature range of collection of carbon black - 1200 - 1300°C.

Carbon black obtained from	Yield %	Ash %	Moisture %	V.M. %	F.C. %	C %	H %	S %	N+O (by diff.)
1. Baragolai colliery, Upper Assam.	20	1.3	2.0	2.2	94.5	95.8	0.7	1.6	1.9
2. Garo Hill, Lower Assam.	22	4.2	2.5	2.6	90.7	95.4	0.8	1.7	2.1
3. Pench Valley, M.P.	14.4	18.3	1.4	3.8	76.5	94.7	0.7	0.4	4.1

Table III - Properties of carbon black obtained from Assam coal compared with those of commercial carbon black.

Carbon black obtained from	Acetone extract %	Iodine absorption values mg./g.	D.B.P. absorption values ml/100g.	Particle size, °A	Surface area M^2/g.	pH
1. Baragolai colliery coal, Upper Assam.	1.9	89	106	200 - 800	68	6.5 to 7.0
2. SRF black	0.4	30	70 - 80	600 - 800	25 - 30	6.0 - 6.5
3. HAF black	0.5	82	103+6 -	200 - 800	74 -100	8.0 - 9.0

Table IV - Physical properties of rubber compounded with *ACB and SRF.

Compound No.	50 phr SRF			50 phr ACB sample			60 phr ACB sample			70 phr ACB sample		
Mooney viscosity (ML4) @ 100°C	63.5			74.5			82.0			95.5		
Mooney scorch (MS) Time @ 127°C.	32'-43"			21'-30"			20'-25"			18'-30"		
Cure Index (RC) @ 127°C	6'-0"			5'-0"			4'-40"			4'-30"		
Time of cure @ 145°C	15'	25'	35'	15'	25'	35'	15'	25'	35'	15'	25'	35'
Tensile strength lb./sq. ft.	2946	2974	3060	2960	2803	2817	2789	2960	2760	2660	2617	2560
Elongation at break %	670	396	385	530	400	400	500	385	350	400	300	290
300% Modulus lb./cu.ft.	1217	2174	2230	1459	1945	1973	1730	2160	2274	2074	2560	-
Shore hardness A_2	61	65	65	68	70	70	70	71	71	77	79	79

*ACB indicates carbon black obtained from Baragolai colliery coal, Upper Assam.

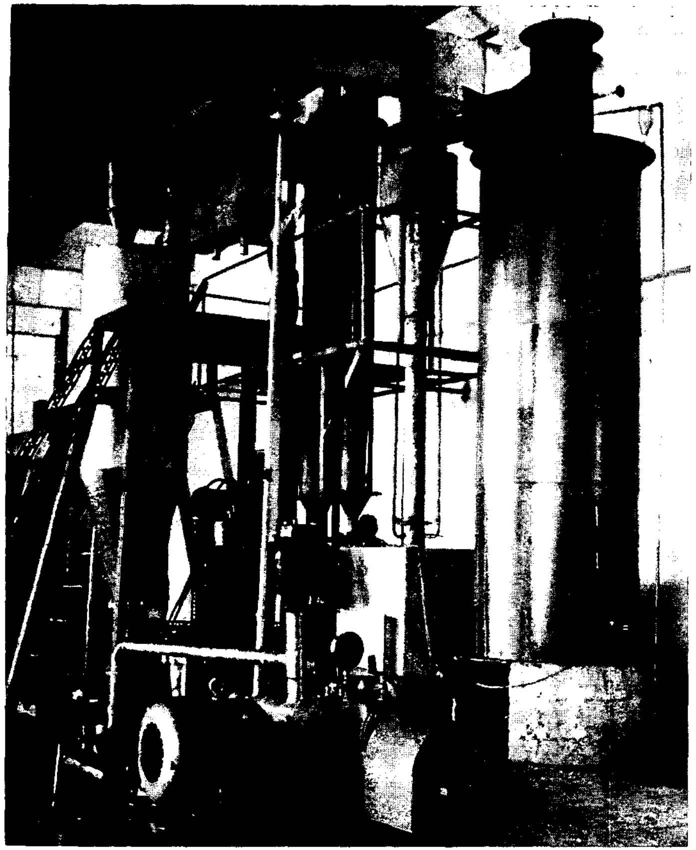


Figure 1. The unit for the production of carbon black from coal.

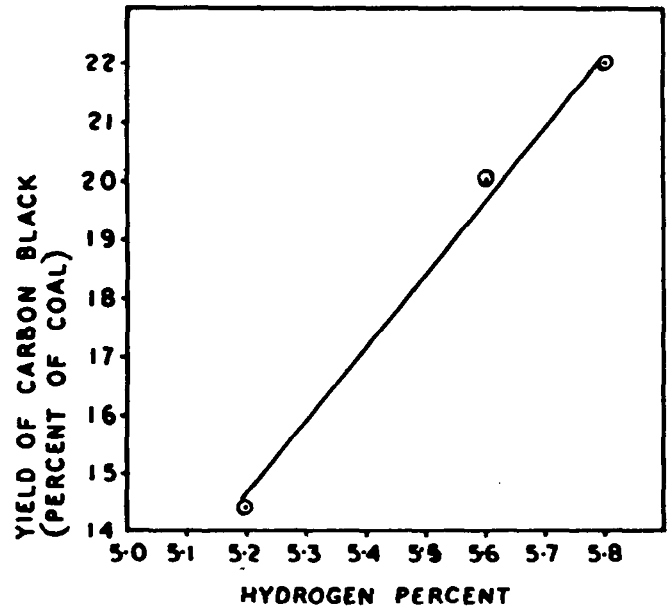


FIGURE-2. RELATIONSHIP BETWEEN HYDROGEN CONTENT OF COAL AND YIELD OF CARBON BLACK FROM IT.

Figure 2. Relationship between hydrogen content of coal and yield of carbon black from it.

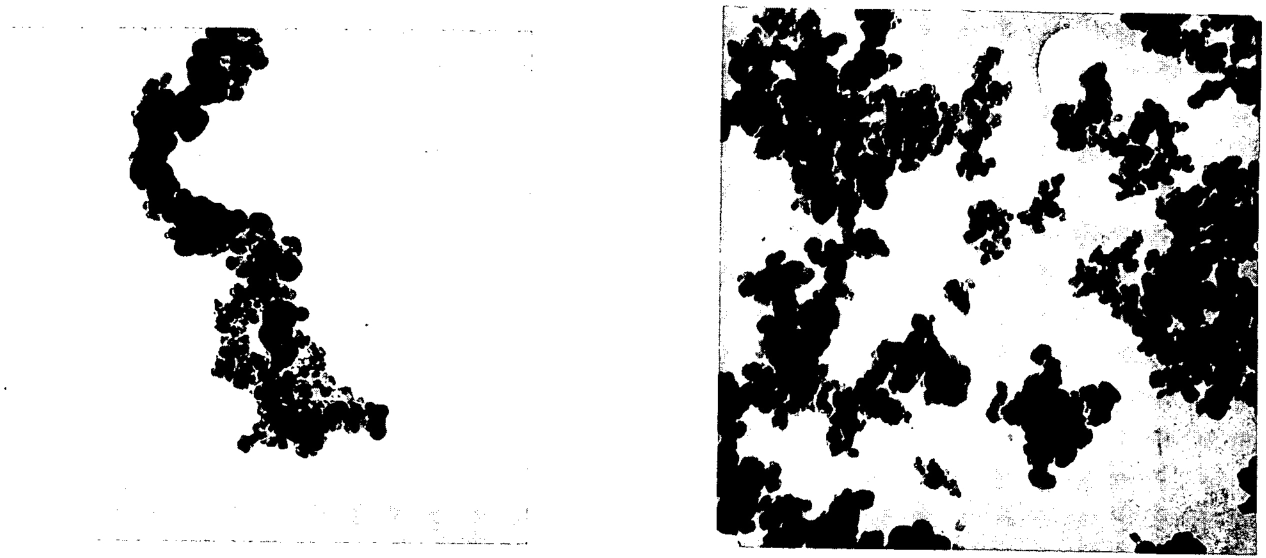
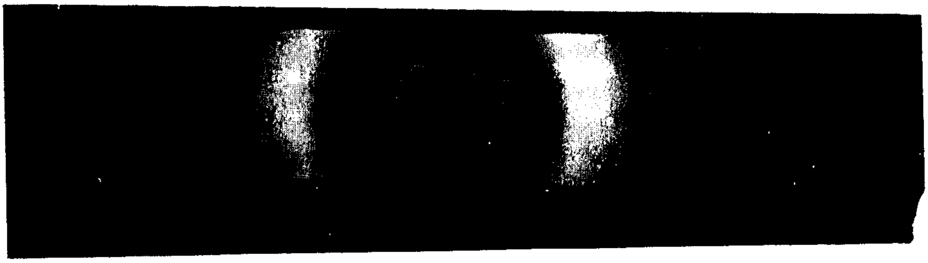
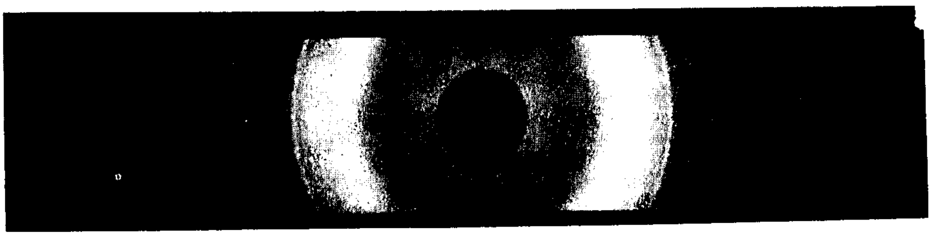


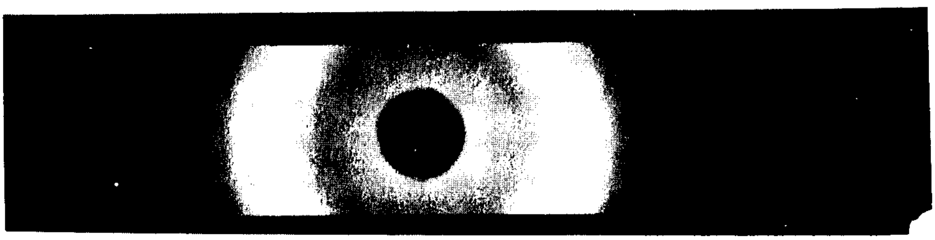
Figure 3. Electron micrograph of carbon blacks produced from (a) Baragolai colliery coal, Upper Assam, (b) HAF black (magnification 52700 times).



(1)



(2)



(3)

Figure 4. X-ray diffraction patterns of the carbon blacks obtained from (1) Baragolai colliery coal, Upper Assam (2) Pench Valley coal, M.P., (3) HAF black.